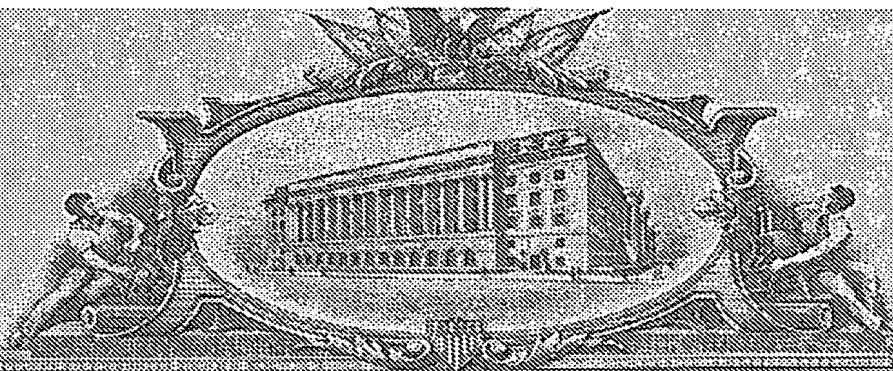1236428

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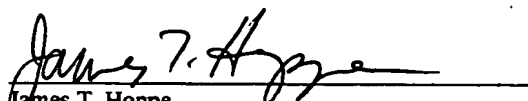Patent and Trademark Office

PROVISIONAL APPLICATION COVER SHEET

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TITLE OF THE INVENTION (280 characters max)

IMPROVED RESIN COMPOSITIONS FOR EXTRUSION COATING

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PROVISIONAL APPLICATION FILING ONLY

IMPROVED RESIN COMPOSITIONS FOR EXTRUSION COATING

Field Of The Invention

This invention pertains to polyethylene extrusion compositions. In
5 particular, the invention pertains to ethylene polymer extrusion compositions
having high drawdown and substantially reduced neck-in. The invention also
pertains to a method of making the ethylene polymer extrusion composition and a
method for making an extrusion coated article, an article in the form of an
extrusion profile and an article in the form of an extrusion cast film.

10

Background and Summary of the Invention

It is known that low density polyethylene (LDPE) made by high-
pressure polymerization of ethylene with free-radical initiators as well as
heterogeneous linear low density polyethylene (LLDPE) and ultra low density
15 polyethylene (ULDPE) made by the copolymerization of ethylene and α−olefins
with Ziegler coordination (transition metal) catalysts at low to medium pressures
can be used, for example, to extrusion coat substrates such as paper board, paper,
and/or polymeric substrates; to prepare extrusion cast film for applications such as
disposable diapers and food packaging; and to prepare extrusion profiles such as
20 wire and cable jacketing. However, although LDPE generally exhibits excellent
extrusion processability and high extrusion drawdown rates, LDPE extrusion
compositions lack sufficient abuse resistance and toughness for many applications.
For extrusion coating and extrusion casting purposes, efforts to improve abuse
properties by providing LDPE compositions having high molecular weights (i.e.,
25 having melt index, I_2, less than about 2 g/10min) are not effective since such
compositions inevitably have too much melt strength to be successfully drawn
down at high line speeds.

While LLDPE and ULDPE extrusion compositions offer improved
abuse resistance and toughness properties and MDPE (medium density
30 polyethylene) extrusion compositions offer improved barrier resistance (against,
for example, moisture and grease permeation), these linear ethylene polymers
exhibit unacceptably high neck-in and draw instability; they also exhibit relatively
poor extrusion processability compared to pure LDPE.

It is generally believed that neck-in and melt strength are inversely
35 related. Thus, in references such as U.S. Patents 5,582,923 and 5,777,155 to Kale

et al. (each of which is hereby incorporated by reference in its entirety), adding LLDPE to improve physical toughness came at the expense of extrudability factors such as increased neck-in. Thus in the extrusion coating industry, current practice is to utilize lower melt index LDPE for extrusion on equipment with narrower die

5   widths and relatively low maximum take off rates. Such low melt index autoclave LDPE resins provide low neck-in (less than about 2 inches (1 inch for each side)) and sufficient draw-down speed. This is typically with older equipment. Faster equipment, typically with wider die widths and improved internal deckling, is supplied with higher melt index autoclave LDPE, which unfortunately, tends to

10   yield greater neck-in. Thus several grades of LDPE are currently required, necessitating complex product handling capability at both resin manufacturer and at the extrusion coating facility, with additional requirements for inventory storage. It would therefore be advantageous to provide a single grade of resin which simultaneously yielded high draw-down and low neck-in.

15   Such a new family of compositions has now been discovered which breaks the traditional relationship between neck-in and melt strength, meaning that the neck-in can be controlled independently of the melt strength of the polymer. This allows the melt strength to be varied over wide ranges without substantially affecting the fabrication performance. Thus substantially the same neck-in is

20   obtained for resins which vary over a wide range of melt indexes. In the preferred practice of the present invention this neck-in is less than approximately two inches (1" per side) at a haul-off rate of approximately 400 feet/minute. Surprisingly, in contrast to pure LLDPE, the neck-in decreases on increasing haul-off rates. The practical range of melt index is from about 3 to about 30 in most coating

25   applications, and the compositions of the present invention can cover this entire range. It is desirable that the maximum operating speed of the extrusion coating equipment not be limited by the properties of the resin being used. Thus it is desirable to use resin which exhibits neither draw instability nor breaking before the maximum line speed is reached. It is even more desirable that such resin

30   exhibit very low neck-in, less than about 2 inches. The resins provided in this invention exhibit low neck-in and excellent draw stability while the draw-down capability required is obtained by selecting the correct melt index. Typically the melt index is in the range of 10-20. It is a further feature of this invention that it provides a resin at for example 15 MI that will be suitable for extrusion on both

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older equipment having slow take-off and modern high speed equipment. In both situations the neck-in can be less than 2 inches.

The resin compositions of the present invention can be characterized by their unique rheological properties. It is believed that the unique

5   rheology of composition of the present invention is required to achieve the independence of the neck-in versus melt index observed with the compositions. The compositions can be characterized as having a rheology such that the estimated slope (or S) (by linear least squares regression) of a natural log (-loss modulus (or G")) versus natural log (storage modulus (or G')) is greater than

10   [0.635*(melt index)+13.2]/[(melt index)+16.6], wherein the melt index is the melt index of the composition measured in accordance with ASTM D1238, condition 190°C/2.16kg.

It has been discovered that the desired rheology is obtained from resins containing relatively small amounts of molecules having high molecular

15   weight and which are highly branched. Resins with this structural component exhibit the surprising combination of low neck-in and high draw down. Accordingly, the resins of the present invention can also be distinguished according to their structural architecture as determined by gel permeation chromatography. The resins of the current invention are those in which the

20   percentage fraction of a GPC chromatogram which has a molecular weight above 85,000 as determined by gel permeation chromatography with a differential refractometer detector, is less than 23% of the total area of the GPC chromatogram, and yet more than 10% of the total light scattering area from a low angle laser light scattering detector occurs above a conventional GPC molecular

25   weight of 1,750,000 atomic units. Further, the branching of the high molecular weight portion of the composition is preferably tree-like as opposed to comb-like as expressed in the Mark-Houwink plot (such as described by Wood-Adams, Dealy, deGroot and Redwine, "Effect of Molecular Structure on the Linear Viscoelastic Behavior of Polyethylene", 33 *Macromolecules 2000*, 7489), where

30   the slope is in the range of 0.1 to 0.25 in the absolute molecular weight range between 300,000 and 3,000,000.

The resin compositions may advantageously comprise a blend of materials such as an LLDPE material together with an LDPE having a high molecular weight, highly branched component. The compositions may also

35   include other polymeric materials such as polypropylene, ethylene-styrene

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copolymers, high pressure ethylene copolymers such as ethylvinylacetate (EVA) and ethylene acrylic acid, and the like.

LLDPE in the preferred blends for use in making the compositions of the present invention can be any LLDPE known in the art, and can be varied
5   depending on the physical properties desired, as is generally known in the art.

The preferred LDPE for use in making the compositions of the present invention has a Melt Index (I2) as measured in accordance with ASTM D1238, condition 190°C/2.16kg of from about 2 to about 0.2 g/10 min, a melt strength of from about 25 to 50 cN or greater respectively, and an Mw/Mn as
10 measured by conventional GPC greater than about 10. Such an LDPE is preferably made in an autoclave reactor with chilled ethylene feed below 35°C operating in single phase mode with three or more zones. The reactor is preferably run above the transition point (phase boundary between a two -phase and a single phase system) at an average reactor temperature of approximately 240°C.
15 Another aspect of the present invention is a process for improving extrusion coating performance by using the resins of the present invention.

DESCRIPTION OF THE DRAWINGS

Fig.1 is a plot of the natural log of loss modulus G" versus elastic or storage modulus G' for a series of resins.
20 Fig. 2 is a plot of the slope of the line obtained from a linear least squares regression from the plot in Figure 1 versus melt index.

Fig. 3 is an example of an appropriate light scattering chromatogram obtained on an NBS 1476 high pressure low density polyethylene standard.
25 Fig. 4 is a plot of the cumulative detector fraction (CDF) of the low angle light scattering detector (LS) versus the log base 10 of the linear polyethylene equivalent GPC MW

Fig. 5 is a plot the cumulative detector fraction (CDF) of the refractometer detector (RI) versus the log base 10 of the linear polyethylene
30 equivalent GPC MW

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The following terms shall have the given meaning for the purposes
35 of this invention:

4

"Haul-Off" is defined herein to mean the speed at which the substrate is moving, thus stretching or elongating a molten polymer extrudate.

"Drawdown" is defined as the haul-off speed at which the molten polymer breaks from the die or the speed at which edge instability was noted.

5   "Melt strength" which is also referred to in the relevant art as "melt tension" is defined and quantified herein to mean the stress or force (as applied by a wind-up drum equipped with a strain cell) required to draw a molten extrudate at a haul-off velocity at which the melt strength plateaus prior to breakage rate above its melting point as it passes through the die of a standard plastometer such as the

10   one described in ASTM D1238-E. Melt strength values, which are reported herein in centi-Newtons (cN), are determined using a Gottfert Rheotens at 190°C. Density is tested in accordance with ASTM D792.

"Neck-in" is defined herein as the difference between the die width and the extrudate width on the fabricated article. The neck-in values reported

15   herein are determined at a haul off rate of 440 feet/minute which yields a 1 mil coating thickness at an extrusion rate of approximately 250 lbs/hr, using a 3.5-inch diameter, 30:1 L/D Black-Clawson extrusion coater equipped with a 30 inch wide die deckled to 24 inches and having a 25-mil die gap.

The term "polymer", as used herein, refers to a polymeric

20   compound prepared by polymerizing monomers, whether of the same or a different type. The generic term polymer thus embraces the term "homopolymer", usually employed to refer to polymers prepared from only one type of monomer as well as "copolymer" which refers to polymers prepared from two or more different monomers.

25   The term "LDPE" may also be referred to as "high pressure ethylene polymer" or "highly branched polyethylene" and is defined to mean that the polymer is partly or entirely homopolymerized or copolymerized in autoclave or tubular reactors at pressures above 14,500 psi (100 MPa) with the use of free-radical initiators, such as peroxides (see for example US 4,599,392, herein

30   incorporated by reference).

The term "LLDPE" is defined to mean any linear or substantially linear polyethylene copolymer. The LLDPE can be made by any process such as gas phase, solution phase, or slurry or combinations thereof.

Description of the Composition

The composition of matter of the present invention can be characterized according to its rheological properties and its molecular architecture as determined by multiple detector GPC.

5   It is believed that the rheology of the composition of the present invention leads to the independence of the neck-in versus melt index of the composition. The rheology of the compositions can be demonstrated according to the following method which uses dynamic shear rheology to independently measure the elastic and viscous responses of a polymer sample. The measurement

10   should be performed at a temperature of 190° C and a range of frequencies from 0.1 to 100 1/sec using an oscillatory shear rheometer such as a Rheometrics RMS-800. The natural logarithm of these values can then be plotted (ln (G") vs. ln(G').

A linear least squares regression can be performed on the ln(G") vs. ln (G') data, resulting in an equation of the form:

15             $Ln(G") = (S)*(\ln(G')) + G$,

Where S is the regressed slope of the line, and G is the natural log of G" when the natural log of G' is zero.

It has been discovered that the S determined by the regression is an especially useful parameter for quantifying the elasticity of each resin.

20   Compositions of the present invention can be differentiated by having an S exceeding [0.635*(melt index)+13.2]/[(melt index)+16.6]. Preferably, the compositions of the present invention exceed this value by more than 1%, more preferably more than 1.5%.

These compositions surprisingly exhibit neck-in performance in

25   extrusion coating which is effectively independent of the melt index. This independence is not achieved at the expense of a reduction in draw-down. Thus it is possible to provide resins having high melt index and high draw down but surprisingly low neck-in.

The composition of matter of the present invention can also be

30   characterized according to its unique molecular architecture as evidenced by multiple detector gel permeation chromatography (GPC). When using a properly calibrated GPC, equipped with at least a low angle laser light scattering detector (LS) and a conventional refractive index (RI) detector, the compositions of the present invention will exhibit the following properties: First, the percentage

35   fraction of a GPC chromatogram which has a linear equivalent polyethylene

molecular weight above 85,000 as determined by gel permeation chromatography with a differential refractometer detector, is less than about 23% of the total area of the GPC chromatogram, and yet more than about 10% of the total light scattering area from a low angle laser light scattering detector occurs above a conventional

5   GPC molecular weight of 1,750,000 atomic units. Preferably, the material will have a percentage fraction of a GPC chromatogram which has a molecular weight above 85,000 as determined by gel permeation chromatography with a differential refractometer detector, less than about 21%, more preferably less than about 20 % and most preferably less than about 15% of the total area of the GPC

10   chromatogram. Preferably the material will also have more than 12% of the total light scattering area from a low angle laser light scattering detector occurs above a conventional GPC molecular weight of 1,750,000 atomic units.

      The compositions of the present invention can have any melt strength, and surprisingly in view of the low neck-in observed with these resins,

15   melt strength less than about 9cN and more preferably less than about 5 cN are particularly advantageous.

      As should be understood from the foregoing, the physical properties of the compositions of the present invention when coated on a substrate can be tailored according to the particular needs. In the preferred blends of the invention,

20   for example, the particular LLDPE (or combinations of LLDPE with other material including other LLDPE) portion can be selected to optimize whatever characteristics are desired. For example tensile properties and abuse resistance will typically exceed those of conventional LDPE. We have also found that heat seal properties and hot tack properties are superior to conventional LDPE, showing

25   lower initiation temperatures and increased seal and hot tack strength.

      It will be appreciated by one of ordinary skill in the art that the composition of the present invention will be slightly modified after conversion into a fabricated article such as a film layer. Another aspect of the invention is therefore a polymeric film layer having a rheology such that the S of a natural log-

30   natural log plot of loss modulus (or G") versus storage modulus (or G') is greater than [0.66*(melt index)+14.0]/[(melt index)+16.6], and wherein the polymeric material has a CDF RI fraction less than about 0.23 of GPC chromatogram which has a linear equivalent polyethylene molecular weight above 85,000, and a CDF LS fraction of more than about 0.10 at a conventional GPC molecular weight of

35   1,900,000 atomic units or greater. The film layer can preferably be from about 0.1

to about 40 mils. The film layers of the present invention may advantageously be subjected to treatment such as flame or corona treatment and printing as is known in the art. Substrates for use with the film layers can be flame treated, corona treated, or coated with various primers to promote adhesion.

5   Surprisingly, the preferred blends of the present invention exhibit processability (as determined by motor load requirements) similar to traditional high pressure low density polyethylene used in extrusion coating and extrusion lamination applications, despite the majority of composition comprising linear low density polyethylene.

10   The inventive composition can advantageously be achieved using a blend of LLDPE and LDPE, optionally with additional polymers such as minor amounts of polypropylene. The preferred blends of the present invention comprise about 70 percent based on the total weight of the composition, of at least one LLDPE composition. More preferably the composition comprises at least about 75

15   percent, still more preferably 80-85 percent of the LLDPE composition. The inventive composition should contain no more than about 95 percent of the LLDPE composition, but preferably no more than about 90 percent, and most preferably no more than about 85 percent of the LLDPE.

        The inventive composition also contains at least 5 percent,

20   preferably at least 10, more preferably about 15-20 percent, based on the total weight of the composition, of at least one high pressure ethylene polymer composition or LDPE. Preferably this component is no more than 30 percent of the total composition, more preferably no more than 25 percent, more preferably no more than 20 percent. It should be understood that the total amount of LLDPE

25   and LDPE does not necessarily have to equal 100%.

        The molecular architecture of the preferred high pressure ethylene polymer composition is believed to be related to the neck-in, and rheological properties of the final composition. Without intending to be bound to theory, it is believed that the LDPE portion of the preferred blends for the present invention

30   can supply high molecular weight, highly branched structure which leads to the unique combination of rheology and molecular architecture. It should be understood, however that the high molecular weight highly branched portion need not come from a high pressure low density resin, and other processes such as those described in WO 02/074816, may be applicable.

35   The preferred LDPE for use in the present invention has a Melt

Index (I2) of from about 0.2 to about 2g/10 min. More preferably the melt index is greater than about 0.25, most preferably more than 0.3g/10min. The melt index is preferably less than about 1 most preferably less than about 0.7g/10 min. The melt strength of the LDPE is preferably about 25 cN or greater, more preferably30 or

5   greater, and most preferably 40 cN or greater. For practical reasons the melt strength is preferably less than about 100 cN. The preferred LDPE also has a Mw/Mn as measured by conventional GPC greater than about 10, alternatively greater than about 12.

Such an LDPE can be made in an autoclave reactor (optionally

10   configured with a series tube reactor) with chilled ethylene feed below 35°C operating in single phase mode with three or more zones. The reactor is preferably run above the transition point (phase boundary between a two-phase and a single-phase system) at an average reactor temperature of approximately 240°C.

The composition of the present invention may also include

15   LDPE/LDPE blends where one of the LDPE resins has a relatively higher melt index and the other has a lower melt index and is more highly branched. The component with the higher melt index can be obtained from a tubular reactor, and a lower MI, higher branched, component of the blend may be added in a separate extrusion step or using a parallel tubular/autoclave reactor in combination with

20   special methods to control the melt index of each reactor, such as recovery of telomer in the recycle stream or adding fresh ethylene to the autoclave (AC) reactor, or any other methods known in the art.

Suitable high pressure ethylene polymer compositions for use in preparing the inventive extrusion composition include low density polyethylene

25   (homopolymer), ethylene copolymerized with at least one α–olefin e.g. butene, and ethylene copolymerized with at least one αβ-ethylenically unsaturated comonomers, e.g., acrylic acid, methacrylic acid, methyl acrylate and vinyl acetate. A suitable technique for preparing useful high pressure ethylene copolymer compositions is described by McKinney et al. in US Patent 4,599,392, the

30   disclosure of which is incorporated herein by reference.

While both high pressure ethylene homopolymers and copolymers are believed to be useful in the invention, homopolymer polyethylene is generally preferred.

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LLDPE

Any type of LLDPE can be used in the blends which make up the preferred compositions of the present invention. This includes the substantially linear ethylene polymers which are further defined in U.S. Patent 5,272,236, U.S.

5   Patent 5,278,272, U.S. Patent 5,582,923 and US Patent 5,733,155; the homogeneously branched linear ethylene polymer compositions such as those in U.S. Patent No. 3,645,992; the heterogeneously branched ethylene polymers such as those prepared according to the process disclosed in U.S. Patent No. 4,076,698; and/or blends thereof (such as those disclosed in US 3,914,342 or US 5,854,045).

10   Each of these references is incorporated herein by reference. The LLDPE can be made via gas-phase, solution-phase or slurry polymerization or any combination thereof, using any type of reactor or reactor configuration known in the art.

When used in the present invention, the LLDPE preferably has a melt index of 15 g/10 min or greater, more preferably greater than 20 and most

15   preferably about 30 g/10min. The melt index of the LLDPE can be as high as about 1000 g/10 min, preferably 500 or less, most preferably less than 50 g/10 min.

Preparation of the Polymer Extrusion Composition

The preferred blends for making the polymer extrusion compositions of this invention can be prepared by any suitable means known in the

20   art including tumble dry-blending, weigh feeding, solvent blending, melt blending via compound or side-arm extrusion, or the like as well as combinations thereof. Surprisingly, in view of the melt index difference between the low melt index LDPE component and the higher melt index component, either LLDPE or LDPE, these blends do not require special mixing procedures to prevent gels. Dry blend

25   mixes extruded with a 30:1 single screw provide a resulting film layer with minimal unmixed gels. Such procedures are often required with fractional melt index tubular resins.

The inventive extrusion composition can also be blended with other polymer materials, such as polypropylene, high pressure ethylene copolymers such

30   as ethylvinylacetate (EVA) and ethylene acrylic acid and the like, ethylene-styrene interpolymers, so long as the necessary rheology and molecular architecture as evidenced by multiple detector GPC are maintained. The composition can be used to prepare monolayer or multilayer articles and structures, for example, as a sealant, adhesive or tie layer. The other polymer materials can be blended with the

10

inventive composition to modify processing, film strength, heat seal, or adhesion characteristics as is generally known in the art.

Both the LDPE and the LLDPE portions of the preferred composition can be used in a chemically and/or physically modified form to
5   prepare the inventive composition. Such modifications can be accomplished by any known technique such as, for example, by ionomerization and extrusion grafting.

Additives such as antioxidants (e.g., hindered phenolics such as Irganox® 1010 or Irganox® 1076 supplied by Ciba Geigy), phosphites (e.g.,
10   Irgafos® 168 also supplied by Ciba Geigy), cling additives (e.g., PIB), Standostab PEPQ™ (supplied by Sandoz), pigments, colorants, fillers, and the like can also be included in the ethylene polymer extrusion composition of the present invention, to the extent that they do not interfere with the high drawdown and substantially reduced neck-in discovered by Applicants. These compositions preferably contain
15   no or only limited amounts of antioxidants as these compounds may interfere with adhesion to the substrate. The article made from or using the inventive composition may also contain additives to enhance antiblocking and coefficient of friction characteristics including, but not limited to, untreated and treated silicon dioxide, talc, calcium carbonate, and clay, as well as primary, secondary and
20   substituted fatty acid amides, chill roll release agents, silicone coatings, etc. Other additives may also be added to enhance the anti-fogging characteristics of, for example, transparent cast films, as described, for example, by Niemann in US Patent 4,486,552, the disclosure of which is incorporated herein by reference. Still other additives, such as quaternary ammonium compounds alone or in combination
25   with ethylene-acrylic acid (EAA) copolymers or other functional polymers, may also be added to enhance the antistatic characteristics of coatings, profiles and films of this invention and allow, for example, the packaging or making of electronically sensitive goods. Other functional polymers such as maleic anhydride grafted polyethylene may also be added to enhance adhesion, especially to polar
30   substrates.

Multilayered constructions comprising the inventive composition can be prepared by any means known including coextrusion, laminations and the like and combinations thereof. Moreover, compositions of this invention can be employed in coextrusion operations where a higher drawdown material is used to
35   essentially "carry" one or more lower drawdown materials. In particular the

compositions of this invention are well suited to carry a material of lower draw-down.

The ethylene polymer extrusion compositions of this invention, whether of monolayer or multilayered construction, can be used to make extrusion
5   coatings, extrusion profiles and extrusion cast films. When the inventive composition is used for coating purposes or in multilayered constructions, substrates or adjacent material layers can be polar or nonpolar including for example, but not limited to, paper products, metals, ceramics, glass and various polymers, particularly other polyolefins, and combinations thereof. For extrusion
10   profiling, various articles can potentially be fabricated including, but not limited to, refrigerator gaskets, wire and cable jacketing, wire coating, medical tubing and water piping, where the physical properties of the composition are suitable for the purpose. Extrusion cast film made from or with the inventive composition can also potentially be used in food packaging and industrial stretch wrap applications.
15

EXAMPLES

A description of all of the resins used in the Examples is presented in Table 1.

Table 1 (Part 1)

Resin	Description	Comonomer	MI g/10min)	Density (g/cc	I10/I	Irganox 101	Irganox 1076	Irgaphos 168
A	Linear polyethylene	None	5	0.96	8	200		1600
B	Linear polyethylene		10	0.96	NM	0	0	0
C	High Pressure LDPE	None	0.47	0.92	NM	300	0	0
D	High Pressure LDPE	None						0
E	Substantially linear ethylene copolym	Octene	3	0.90	8	0	750	0
F	Substantially linear ethylene copolym	Octene	30	0.90	7	0	500	0
G	Substantially linear ethylene copolym	Octene	6	0.90	8	0	500	0
H	High Pressure LDPE	None						0
I	Linear ethylene copolymer	Octene	6	0.92	7.6	200	0	0
J	Linear ethylene copolymer	Octene	25	0.92	NM	0	0	0
K	Substantially linear ethylene copolym	Octene	30	0.90	7	0	0	0
L	Linear ethylene copolymer	Butene	20	0.92	NM	NM	NM	0
M	Substantially linear ethylene copolym	Octene	25	NM	NM	NM	NM	0
N	High Pressure LDPE	None	0.22	0.92	NM	0	0	0
O	High Pressure LDPE	None	2	0.92	NM	0	350	0
P	High Pressure LDPE	None	7	0.92	NM	0	500	0

Blends	Component1	% Compone	Component 2	% Component 2
B1	F	80	C	20
B2	F	40	H	60

NM = Not measured

13

Table 1 (Part 2)

Resin	Irganox 1076	Irgaphos 168	PEPQ	DHT-4A	Erucamide	Process	Catalyst
A		1600	0	0	0	Solution	
B	0	0	0	0	0	Solution	
C	0	0	0	0	0	Autoclave	FreeRadical
D		0			0	Autoclave	FreeRadical
E	750	0	1200	0	0	Solution	Constrained Geometry
F	500	0	800	0	600	Solution	Constrained Geometry
G	500	0	800	0	0	Solution	Constrained Geometry
H		0				Autoclave	FreeRadical
I	0	0	0	750	0	Solution	Ziegler-Natta
J	0	0	0	0	0	Solution	Ziegler-Natta
K	0	0	0	0	0	Solution	Constrained Geometry
L	NM	0	NM	NM	0	Gas phase	Ziegler/Natta
M	NM	0	NM	NM	NM	Solution	Constrained Geometry/Ziegler-Natta
N	0	0	0	0	0	Tube	FreeRadical
O	350	0	0	0	0	Tube	FreeRadical
P	500	0	0	0	0	Tube	FreeRadical

Rheology Determination

   To illustrate a suitable process for determining the rheology of polymeric material, samples of Resins A, B, C, D, E, B1 and B2 were prepared.
5 The loss modulus G" and the elastic or storage modulus G' for each of these compositions was then determined by Rheometrics RMS800 oscillatory shear rheometer. The values obtained for Resin B1, Resin D and Resin A are reported in Table 2 and a natural log plot of these values (along with the plots for additional resins) is shown in Figure 1.

10

Resin B1: Blend composition 14.7 MI, 0.9127 g/cc				
S = .7606, G = 3.431				
Freq	G'	G"	ln G'	ln G"
rad/s	dyn/cm2	dyn/cm2		
0.1	51.2707	711.76	3.937119	6.567741
0.15849	100.718	1101.21	4.612325	7.004165
0.25119	190.409	1696.52	5.249174	7.436334
0.39811	351.206	2591.23	5.861373	7.859888
0.63096	631.134	3921.32	6.447518	8.274184
1	1096.95	5888.39	7.000289	8.680738
1.58489	1862.58	8773.37	7.529718	9.079476
2.51189	3083.53	12952.7	8.03383	9.46906
3.98107	5011.2	18971.9	8.519431	9.850714
6.30957	8007.38	27555.7	8.988119	10.22396
10	12603.8	39747.5	9.441754	10.5903
15.8489	19593.4	56902	9.882948	10.94909
25.1189	30172.2	80901.4	10.31468	11.30099
39.8107	45941.2	1.14E+05	10.73512	11.64308
63.0957	69399.6	1.59E+05	11.14764	11.9759
100	1.04E+05	2.19E+05	11.55445	12.29628

Resin D: LDPE 4.15 MI, 0.924 g/cc
$S = .7281$, $G = 3.256$

Freq rad/s	G' dyn/cm2	G" dyn/cm2	ln G'	ln G"
0.1	938.84	4103.7	6.844645	8.319644
0.1585	1678.3	6.00E+03	7.425537	8.699715
0.2512	2886.8	8.57E+03	7.967904	9.055895
0.3981	4.73E+03	1.20E+04	8.461955	9.392662
0.631	7.49E+03	1.66E+04	8.921511	9.717158
1	1.15E+04	2.24E+04	9.350102	10.01682
1.585	1.70E+04	2.99E+04	9.740969	10.30561
2.512	2.46E+04	3.94E+04	10.1105	10.58152
3.9813	3.48E+04	5.11E+04	10.45737	10.84154
6.3101	4.84E+04	6.55E+04	10.78726	11.08981
10.001	6.61E+04	8.31E+04	11.09892	11.3278
15.85	8.89E+04	1.04E+05	11.39527	11.55215
25.121	1.18E+05	1.30E+05	11.67844	11.77529
39.813	1.54E+05	1.59E+05	11.94471	11.97666
63.101	1.99E+05	1.94E+05	12.20106	12.17561
100	2.53E+05	2.34E+05	12.44114	12.36308

Resin A: HDPE 5.162 MI, 0.9625 g/cc
$S = .7174$, $G= 4.16$

| Freq rad/s	G' dyn/cm2	G" dyn/cm2	ln G'	ln G"
0.1	123.62	2180.5	4.817212	7.687309
0.1585	234.09	3272	5.455706	8.093157
0.2512	462.24	5074.2	6.136084	8.531924
0.3981	852.31	7.80E+03	6.74795	8.961481
0.631	1524.4	1.20E+04	7.329356	9.392662
1	2689.6	1.82E+04	7.897148	9.809177
1.585	4795.4	2.75E+04	8.475412	10.22194
2.512	8.26E+03	4.11E+04	9.019737	10.62376
3.9813	1.42E+04	6.09E+04	9.560997	11.01699
6.3101	2.41E+04	8.93E+04	10.08997	11.39976
10.001	4.00E+04	1.30E+05	10.59663	11.77529
15.85	6.54E+04	1.85E+05	11.08828	12.12811
25.121	1.05E+05	2.60E+05	11.56172	12.46844
39.813	1.65E+05	3.59E+05	12.0137	12.79108
63.101	2.54E+05	4.86E+05	12.44509	13.09396
100	3.81E+05	6.43E+05	12.85055	13.3739
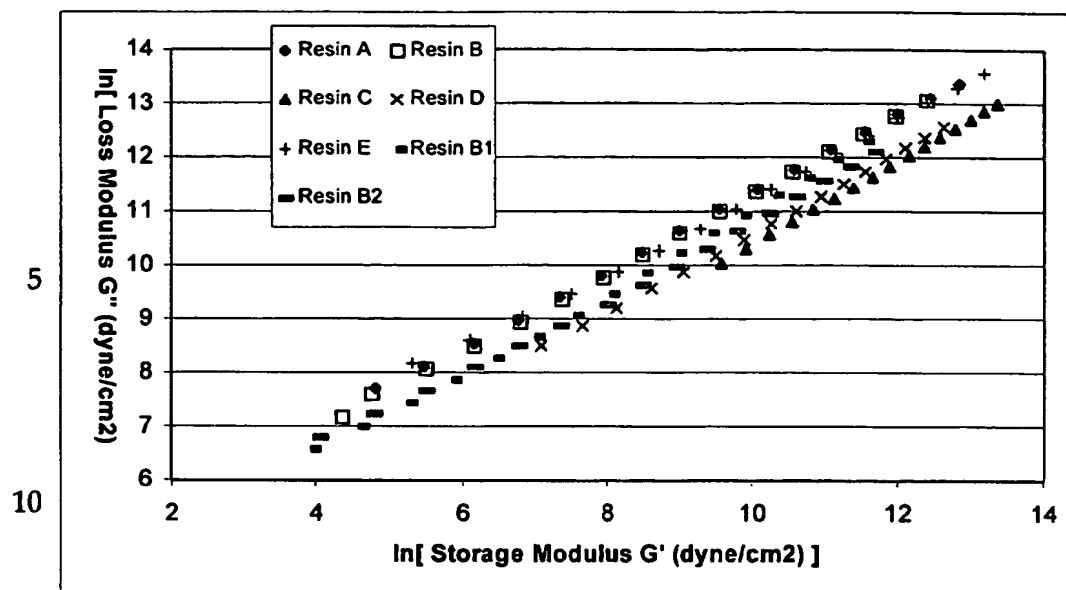
A linear least squares regression can be performed on the ln (G") vs. ln (G') data for each of the resins, resulting in an equation of the form:

ln (G") = (S)*(ln (G') + G; Where S is the regressed slope of the line and G is the natural log of G" when the natural log of G' is zero.

Values for this slope can then be plotted against the melt index. These values are shown in the plot below.

The line formed by the equation S = [0.635*(melt index)+13.2]/[(melt index)+16.6] is also presented on this plot, and those compositions which have slopes below this line are not within the scope of the present invention.
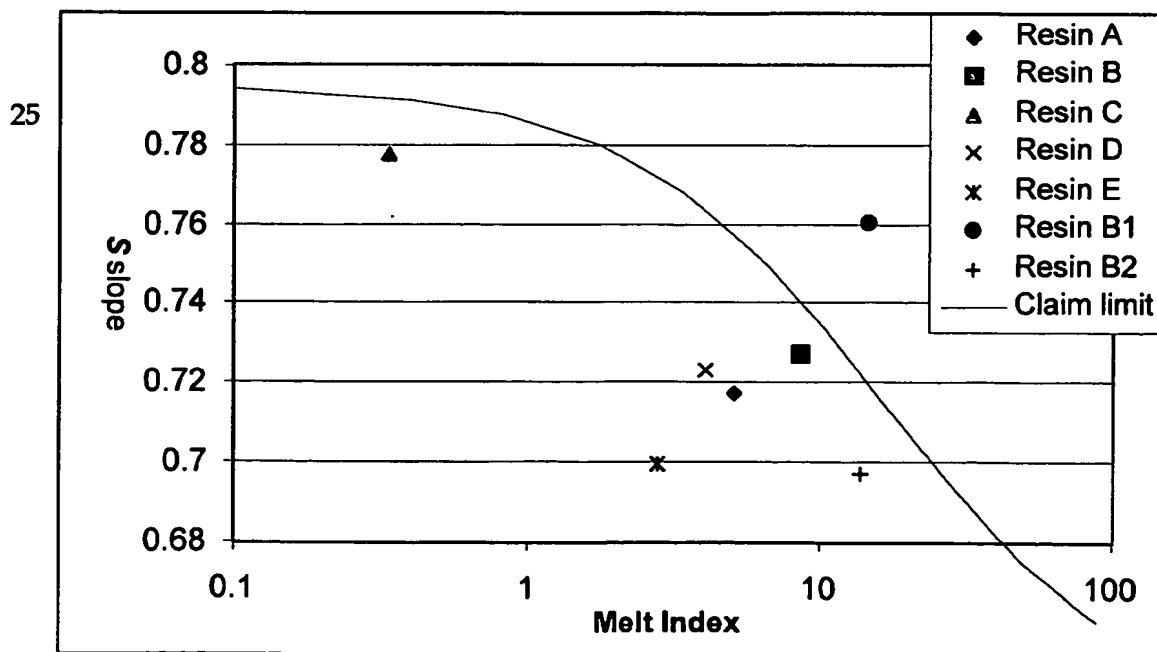
Molecular Architecture Determination

    In order to determine the molecular architecture of various polymer
5 compositions, the following procedure was used:
    The chromatographic system consisted of a Waters (Millford, MA)
150C high temperature chromatograph equipped with a Precision Detectors
(Amherst, MA) 2-angle laser light scattering detector Model 2040. The 15-degree
angle of the light scattering detector was used for the calculation of molecular
10 weights. Data collection was performed using Viscotek TriSEC software version 3
and a 4-channel Viscotek Data Manager DM400. The system was equipped with
an on-line solvent degas device from Polymer Laboratories.

    The carousel compartment was operated at 140°C and the column
15 compartment was operated at 150°C. The columns used were 7 Polymer
Laboratories 20-micron Mixed-A LS columns. The solvent used was 1,2,4
trichlorobenzene. The samples were prepared at a concentration of 0.1 grams of
polymer in 50 milliliters of solvent. The chromatographic solvent and the sample
preparation solvent contained 200 ppm of butylated hydroxytoluene (BHT). Both
20 solvent sources were nitrogen sparged. Polyethylene samples were stirred gently at
160 degrees Celsius for 4 hours. The injection volume used was 200 microliters
and the flow rate was 1.0 milliliters/minute.

    Calibration of the GPC column set was performed with 18 narrow
25 molecular weight distribution polystyrene standards with molecular weights
ranging from 580 to 8,400,000 and were arranged in 5 "cocktail" mixtures with at
least a decade of separation between individual molecular weights. The standards
were purchased from Polymer Laboratories (Shropshire, UK). The polystyrene
standards were prepared at 0.025 grams in 50 milliliters of solvent for molecular
30 weights equal to or greater than 1,000,000, and 0.05 grams in 50 milliliters of
solvent for molecular weights less than 1,000,000. The polystyrene standards were
dissolved at 80 degrees Celsius with gentle agitation for 30 minutes. The narrow
standards mixtures were run first and in order of decreasing highest molecular
weight component to minimize degradation. The polystyrene standard peak
35 molecular weights were converted to polyethylene molecular weights using the

following equation (as described in Williams and Ward, J. Polym. Sci., Polym. Let., 6, 621 (1968)).:

$$M_{polyethylene} = A \times (M_{polystyrene})^B$$

5   *Where M is the molecular weight, A has a value of 0.41 and B is equal to 1.0.*

A fourth order polynomial was used to fit the respective polyethylene-equivalent calibration points.

10

The total plate count of the GPC column set was performed with Eicosane (prepared at 0.04 g in 50 milliliters of TCB and dissolved for 20 minutes with gentle agitation.) The plate count and symmetry were measured on a 200 microliter injection according to the following equations:

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    PlateCount = 5.54 * (RV at Peak Maximum / (Peak width at ½ height)) ^ 2

    Where RV is the retention volume in milliliters and the peak width is in milliliters.

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    Symmetry = (Rear peak width at one tenth height - RV at Peak maximum) / (RV at Peak Maximum - Front peak width at one tenth height)

    Where RV is the retention volume in milliliters and the peak width is in
25 *milliliters.*

The Systematic Approach for the determination of multi-detector offsets was done in a manner consistent with that published by Balke, Mourey, et. Al (Mourey and Balke, Chromatography Polym. Chpt 12, (1992)) (Balke,
30 Thitiratsakul, Lew, Cheung, Mourey, Chromatography Polym. Chpt 13, (1992)), optimizing dual detector log MW results from Dow broad polystyrene 1683 to the narrow standard column calibration results from the narrow standards calibration curve using in-house software. The molecular weight data was obtained in a manner consistent with that published by Zimm (Zimm,B.H., J.Chem. Phys., 16,
35 1099 (1948)) and Kratochvil (Kratochvil, P., *Classical Light Scattering from*

Polymer Solutions, Elsevier, Oxford, NY (1987)). The overall injected
concentration used for the determination of the molecular weight was obtained
from the sample refractive index area and the refractive index detector calibration
from a linear polyethylene homopolymer of 115,000 molecular weight. The
5   chromatographic concentrations were assumed low enough to eliminate addressing
2nd Virial coefficient effects (concentration effects on molecular weight).

     In order to monitor the deviations over time, which may contain an elution
component (caused by chromatographic changes) and a flowrate component
10   (caused by pump changes), a late eluting narrow peak is generally used as a
"marker peak". A flowrate marker was therefore established based on the air peak
mismatch between the degassed chromatographic system solvent and the elution
sample on one of the polystyrene cocktail mixtures. This flowrate marker was used
to linearly correct the flowrate for all samples by alignment of the air peaks. Any
15   changes in the time of the marker peak are then assumed to be related to a linear
shift in both flowrate and chromatographic slope.

     To facilitate the highest accuracy of a retention volume (RV) measurement
of the flow marker peak, a least-squares fitting routine is used to fit the peak of the
20   flow marker concentration chromatogram to a quadratic equation. The first
derivative of the quadratic equation is then used to solve for the true peak position.
After calibrating the system based on a flow marker peak, the effective flowrate (as
a measurement of the calibration slope) is calculated as Equation 1. In a high-
temperature SEC system, an antioxidant mismatch peak or an air peak (if the
25   mobile phase is sufficiently degassed) can be used as an effective flow marker.
The primary features of an effective flowrate marker are as follows: the flow
marker should be mono-dispersed. The flow marker should elute close to the total
column permeation volume. The flow marker should not interfere with the
chromatographic integration window of the sample.
30

     Equation 1 $Flowrate_{effective} = Flowrate_{nominal}$ *
$FlowMarker_{Calibration} / Flowmarker_{Observed}$
     The preferred column set is of 20 micron particle size and "mixed" porosity
to adequately separate the highest molecular weight fractions appropriate to the
35   claims.

The verification of adequate column separation and appropriate shear rate can be made by viewing the low angle (less than 20 degrees) of the on-line light scattering detector on an NBS 1476 high pressure low density polyethylene standard. The appropriate light scattering chromatogram should appear bimodal

5 (very high MW peak and moderate molecular weight peak) with approximately equivalent peak heights as shown in Figure 3. There should be adequate separation by demonstrating a trough height between the two peaks less than half of the total LS peak height. The plate count for the chromatographic system (based on eicosane as discussed previously) should be greater than 32,000 and symmetry

10 should be between 1.00 and 1.12.
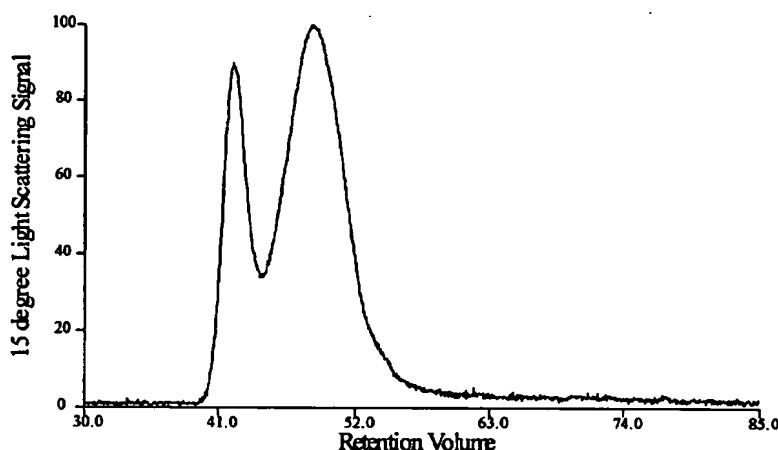
The calculation of the cumulative detector fractions (CDF) for the refractometer

15 ("CDF RI") and the light scattering detector ("CDF LS") are accomplished by the following steps:

1) Linearly flow correct the chromatogram based on the relative retention volume ratio of the air peak between the sample and that of a consistent narrow

20 standards cocktail mixture.

2) Correct the light scattering detector offset relative to the refractometer as described in the calibration section.

3) Subtract baselines from the light scattering and refractometer chromatograms and set integration windows making certain to integrate all of the low

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molecular weight retention volume range in the light scattering chromatogram that is observable from the refractometer chromatogram.

4) Calculate the molecular weights at each data slice based on the polystyrene calibration curve, modified by the polystyrene to polyethylene conversion factor (0.41) as described in the calibration section.

5) Calculate the cumulative detector fraction (CDF) of each chromatogram (CDF RI and CDF LS) based on its baseline-subtracted peak height (H) from high to low molecular weight (low to high retention volume) at each data slice (i) according to the following equation:

$$CDF_i = \frac{\sum\limits_{j=LowestRVindex}^{i} H_j}{\sum\limits_{j=LowestRVindex}^{HighestRVindex} H_j}$$

where i is between the *LowestRVindex and the HighestRVindex*

6) A plot of CDF versus molecular weight is obtained by calculating the CDF at each integrated data slice from step (5) and plotting it versus the log of the polyethylene equivalent molecular weight at each integrated data slice from step (4).

Using this GPC method, an analysis was done on the following blends: a blend comprising 90% Resin F and 10% Resin C; a blend comprising 50% Resin F and 50% Resin C; and a blend comprising 80% Resin G and 20% Resin N. The plots for these materials are shown in Figure 4 and Figure 5. These figures also indicate the log of 85,000 MW for the CDF RI plot, and the log of 1,750,000 MW for CDF LS plot. From these lines it can clearly be seen that the blend of 80% Resin G and 20 % Resin N does not meet the CDF LS criteria for the present invention and neither the blend of 80% Resin G and 20 % Resin N nor the blend comprising 50% Resin F and 50% Resin C meets the CDF RI criteria for the present invention.

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Figure 4

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Low Angle Laser Light Scattering CDF Overlay

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30   Figure 5

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Refractometer CDF Overlay

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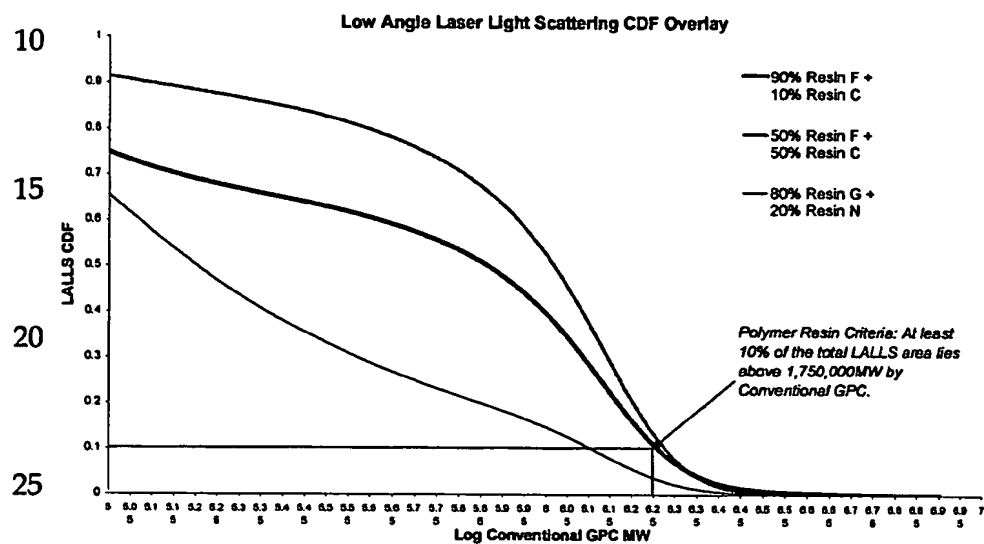
In order to demonstrate the neck-in performance at varying melt
strengths, a series of compositions comprising blends of LLDPE and LDPE were
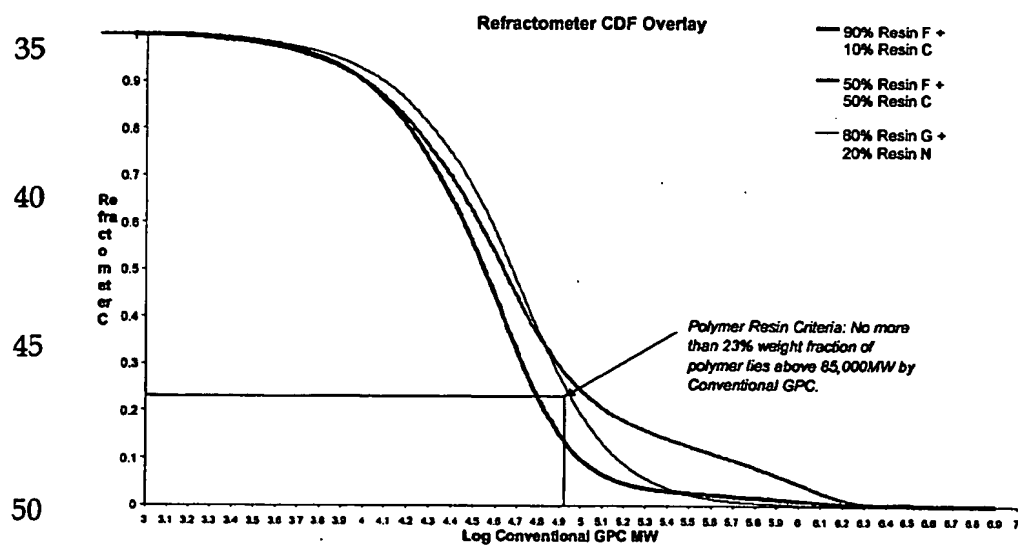prepared. The particular resins and the amounts used are indicated in Table 3.

5   Table 3 also presents the melt strength of the overall blend, the melt index of the
overall blend, the neck-in observed when running at 440 ft/minute at an extrusion
rate of approximately 250 lbs/hr (and where available at 880 ft/min), the drawdown
(a "+" indicates that the drawdown limit was not reached), the slope S determined
in accordance with the rheological methods described above for the unextruded

10   resin, and the CDF RI (expressed as the fraction above 85,000 MW) and CDF LS
(expressed as the fraction above 1,750,000 MW) from the GPC. The table also
reports some determinations of S made on a film layer after extrusion. To obtain
the extruded resin, the extruded resin may be separated from a coated substrate or
more conveniently may be extruded directly onto a suitable metal sheet. This latter

15   method can preferably be done at full extrusion rate (for example 250 lbs/hr) prior
to positioning the die over the substrate intended for the coating operation.

The results clearly show the superiority of the resins of the present invention in
extrusion coating applications.

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5　Table 3 (Part 1)

EXAMPLE	LLDPE	LDPE	% "LLDPE	% "LDPE	MI of Blend	original resin slope S	extrudate slope S	extrudate melt index	CDF RI	CDF LS
1a	RESIN F	RESIN C	100%	0%	N/A	0.643				
1b			90%	10%	22.17	0.706			0.132	0.121
1c			85%	15%	18.70	0.729			0.143	0.123
1d			80%	20%	14.67	0.761	0.811	8.57	0.172	0.125
1e			80%	20%			0.854	10.35		
1f			50%	50%	3.73	0.801			0.277	0.141
1g			40%	60%	2.57	0.803			0.314	0.146
1h			25%	75%	1.29	0.799			0.369	0.153
1i			15%	85%	0.75	0.793			0.406	0.158
1j										
2a	RESIN E	RESIN C	100%	0%	N/A	0.699				
2b			90%	10%	2.21	0.746			0.313	0.101
2c			85%	15%	1.97	0.763			0.321	0.118
2d										
2e										
3a	RESIN F	RESIN H	90%	10%	27.50	0.646			0.117	0.025
3b			85%	15%	25.59	0.649			0.127	0.031
3c			80%	20%	24.67	0.666			0.138	0.034
3d			50%	50%	16.03	0.694			0.202	0.045
3e			40%	60%	13.95	0.697			0.224	0.046
3f			25%	75%	11.41	0.711			0.256	0.048
3g			15%	85%	9.78	0.713			0.277	0.049
3h			100%	0%	N/A	0.643				
3i										
4a	RESIN E	RESIN H	100%	0%	N/A	0.699				
4b			90%	10%	2.90	0.717			0.299	0.042
4c			85%	15%	2.63	0.722			0.300	0.045
4d			80%	20%	2.68	0.728			0.301	0.046
4e			50%	50%	3.34	0.743			0.304	0.049
4f			40%	60%	3.88	0.738			0.305	0.049
4g			25%	75%	4.79	0.735			0.306	0.049
4h			15%	85%	5.70	0.728			0.308	0.049

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Table 3 (Part 2)

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EXAMPLE	Melt Strength of Blend	440 Neck-in (in)	880 Neck-in (in)	Drawdown (ft/min)
1a	0.5	8.25	N/A	850 (Edge Weave)
1b	1	3.5	2.875	1500+
1c	2.7	2.5	2	1500+
1d	3.5	2	1.875	1500+
1e				
1f	15	1.25	N/A	658
1g	16.8	N/A	N/A	350
1h	22.7	N/A	N/A	320
1i	27.5	N/A	N/A	251
1j				
2a	2	7	N/A	550 (DRAW RESONANCE)
2b	7.3	3.375	3.58	1220
2c	10	2.5	N/A	700
2d				
2e				
3a	0.25	8.125	9	950 (EDGEWEAVE) 1100 (Draw resonance
3b	0.5	7.75	6.75	1500+
3c	1	7.625	5.75	1500+
3d	2.1	4	2.625	1500+
3e	4.5	3.125	2.25	1500+
3f	5.7	2.375	1.875	1500+
3g	6.5	2	1.5	1500+
3h	6.5	1.5	1.25	1300
3i				
4a	6.5	1.5	1.25	1300
4b	4	5.125	4.875	1500+
4c	5	4.375	4.125	1500+
4d	5.82	3.75	3.5	1500+
4e	9.7	2.375	2.5	1300
4f	10.4	2.25	2	1250
4g	9.7	2	1.875	1100
4h	9.4	1.75	1.5	1000

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Table 3 (Part 3)

EXAMPLE	LLDPE	LDPE	% "LLDPE	% "LDPE	MI of Blend	original resin slope S	extrudate slope S	extrudate melt inde	CDF RI	CDF LS
5	RESIN G	RESIN N	0.8	0.2	3.22	0.795789249			0.148	0.027
6	RESIN G	RESIN O	0.6	0.4	3.39	0.717086163				
7	RESIN G	RESIN P	0.5	0.5	5.52	0.673375775			0.147	0.039
8	RESIN I		1				0.820036494	2.48		
9a	RESIN K	RESIN C	80	20			0.836326942	7.465	0.172	0.125
9b			80	20			0.812501448	11.12	0.172	0.125
9c			80	20					0.172	0.125
10	RESIN M	RESIN C	85	15			0.875183376			
11a	RESIN L	RESIN C	100	0	20					
11b			90	10	13.7					
11c			80	20	9.4					
12a	RESIN J	RESIN C	100	0	25					
12b			90	10	17				0.134	0.122
12c			85	15	14				0.152	0.135
12d			80	20	11				0.169	0.143
12e			75	25	9				0.187	0.148
12f			70	30	7.6				0.204	0.152

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Table 3 (Part 4)

EXAMPLE	Melt Strength of Blend	440 Neck-in (in)	880 Neck-in (in)	Drawdown (ft/min)
5	6.1	4	3.75	1500+
6	4.5	5.25	4.625	1500+
7	2.7	7	6.75	1500+
8		7		575 D.RES
9a		1.875		
9b		2		
9c		1.75		
10		2 1/8		1450
11a		8 1/8	9 1/4	740(EW)
11b		2 5/8	2 1/2	1500+
11c		1 5/8		740
12a		8.750	10.125	1550 Draw Res
12b		4.250	3.250	2150
12c		2.500	2.250	1800
12d		2.375	2.125	1507
12e		1.875	1.625	1410
12f		1.750	1.625	1080
		2.125	1.875	1200

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We Claim:

1. A composition comprising a polymeric material having a rheology such that the S of a plot of the natural log of loss modulus (or G") versus natural log of storage modulus (or G') is greater than [0.635*(melt index)+13.2]/[(melt index)+16.6], and wherein the polymeric material has a CDF RI fraction less than 0.23 of a GPC chromatogram which has a molecular weight above 85,000 g/mol, and a CDF LS fraction of more than 0.10 at a conventional GPC molecular weight of 1,750,000 g/mol or greater.

2. The composition of Claim 1 wherein the polymeric material has a melt strength less than about 5 cN.

3. The composition of Claim 1 wherein the polymeric material comprises LDPE.

4. The composition of Claim 1 wherein the polymeric material comprises a blend of at least two polymeric materials.

5. The composition of Claim 1 wherein the polymeric material comprises LLDPE.

6. The composition of Claim 3 wherein the LDPE comprises a high molecular weight highly branched component.

7. The composition of Claim 6 wherein the LDPE is made in an autoclave reactor with chilled ethylene feed below 35°C operating in single phase mode.

8. The composition of Claim 1 wherein the polymeric material has a melt index greater than 10 g/10min.

9. The composition of Claim 8 wherein the polymeric material has a melt index greater than about 13.

10. The composition of Claim 8 wherein the polymeric material has a melt index less than about 100.

11. The composition of Claim 1 wherein the polymeric material has a Mark-Houwink plot where the slope is in the range of 0.1 to 0.25 in the absolute molecular weight range between 300,000 and 3,000,000 g/mol.

12. The composition of Claim 1 wherein the value for S is at least 1% greater than [0.635*(melt index)+13.2]/[(melt index)+16.6].

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13. The composition of Claim 1 wherein the polymeric material has a CDF RI fraction less than 0.21 of a GPC chromatogram which has a molecular weight above 85,000 g/mol.

14. The composition of Claim 1 wherein the polymeric material has a CDF RI fraction less than 0.20 of a GPC chromatogram which has a molecular weight above 85,000 g/mol.

15. The use of a composition according to Claim 1 to make a cast film, profile extrusion, or coated substrate.

16. In a process for extruding a polymeric material onto a substrate, the improvement comprising: using a polymeric material having a rheology such that the estimated slope (or S) (by linear least squares regression) of a natural log-natural log plot of loss modulus (or G") versus storage modulus (or G') is greater than [0.635*(melt index)+13.2]/[(melt index)+16.6].

17. The process of Claim 16 wherein the neck-in observed is less than 2 inches when running at a line speed of 440 ft/min.

18. The process of Claim 17 wherein the polymeric material has a melt strength of less than 5 cN.

19. A polymeric film layer having a rheology such that the S of a natural log-natural log plot of loss modulus (or G") versus storage modulus (or G') is greater than [0.66*(melt index)+14.0]/[(melt index)+16.6], and wherein the polymeric material has a CDF RI fraction less than 0.23 of a GPC chromatogram which has a molecular weight above 85,000 g/mol, and a CDF LS fraction of more than 0.10 at a conventional GPC molecular weight of 1,900,000 g/mol or greater.

20. The film layer of Claim 19 in which the film layer was produced using an extrusion coating, extrusion lamination, or cast film process.

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ABSTRACT OF THE DISCLOSURE

        A composition is disclosed which is particularly suited for use in extrusion coating. The composition comprises a polymeric material having a rheology such that the slope S of a natural log-natural log plot of loss modulus (or

5   G") versus storage modulus (or G') is greater than [0.635*(melt index)+13.2]/[(melt index)+16.6], and wherein the polymeric material has a CDF RI fraction less than 0.23 of a GPC chromatogram which has a molecular weight above 85,000 g/mol, and a CDF LS fraction of more than 0.10 at a conventional GPC molecular weight of 1,750,000 g/mol or greater. The compositions exhibit

10   reduced neck-in when used in extrusion coating and the neck-in is independent of melt strength, thereby facilitating improved extrusion processes.

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